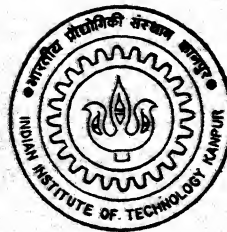


BALLOON COMPRESSION MOULDING = A NEW TECHNIQUE FOR FABRICATING FRP PRODUCTS

by
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March, 1996

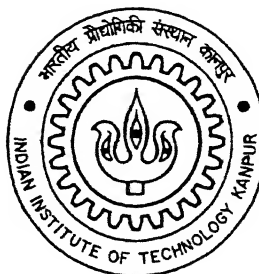
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BALLOON COMPRESSION MOULDING - A NEW TECHNIQUE FOR FABRICATING FRP PRODUCTS

A Thesis Submitted
in Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

by
UDAY M. KOTHALIKAR



to the
**Department of Mechanical Engineering
INDIAN INSTITUTE OF TECHNOLOGY**
March, 1996

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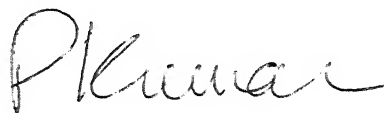
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CERTIFICATE

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Uday M. Kothalikar

It is certified that the work contained in the thesis entitled " **BALLOON COMPRESSION MOULDING - A NEW TECHNIQUE FOR FABRICATING FRP PRODUCT** ", by **Uday M. Kothalikar**, has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.



(Prof. Prashant Kumar)

Department of Mechanical Engineering
Indian Institute of Technology, Kanpur

March, 1996

Dedicated affectionately to

My Parents

and

Sisters

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ABSTRACT

A new FRP fabricating technique, **Balloon Compression Moulding** has been developed to overcome some of limitations of conventional FRP fabrication techniques. Three different products and required tooling has been developed. The products developed are; Circular ring, Convex-concave surfaced ring and Circular ring with concave cross-section (double curvature). The circular ring and convex-concave surfaced ring fabricated with E-class glass fabric and Epoxy resin, using this technique were analyzed for dimensional stability and were found dimensionally stable. Scanning electron microscopy of these components showed good wetting and very few voids were observed. For characterization of circular ring, fibre volume fraction, flexure strength and modulus of elasticity determined. The average flexure strength and average modulus of elasticity obtained were 249.36 Mpa and 20.89 Gpa, respectively at average fibre volume fraction of 39.69%.

CHAPTER 1

INTRODUCTION

1.1 Composite Materials

Composite material is the one, in which two or more distinct materials are embedded in a continuous matrix. The constituent materials have physical and mechanical properties significantly different. The resulting material is one, having properties not attainable by either of these constituent materials existing alone (Agarwal 1990).

The discontinuous material or phase, which is strong & hard and which affects the mechanical properties of the resulting material, is called reinforcing material, continuous phase being called matrix. Usually the reinforcing material is in the form of

continuous or discontinuous fibres, staple or particulate in nature. Matrix can either be metal or polymer compound. We will focus our attention on polymer matrix fibre composite materials.

1.2 Polymer Matrix Fibre Composite Materials

Polymer composite offers attractive properties such as high strength, high stiffness and light weight, compared with conventional materials. Moreover one can tailor make the material as per the design requirements.

It has been observed that, due to a phenomenon called size effect, a small cross-sectioned fibres (5-25 micrometer in diameter) exhibit much higher strength along their longitudinal axis than the strength of bulk material (Chawala 1987). The strength and stiffness of the composite material is greatly influenced by the length of fibres. This is because, the load is not applied directly to the fibres, but to the matrix material and is transferred to the fibres through fibre ends. When the length of fibres is much greater than the length over which stress transfer takes place, the end effects can be neglected.

The fibres being rather small and flexible, can not be directly used, necessitating them to be embedded with a matrix material. S and E-Glass, Carbon, Kevlar and Boron fibres are the most commonly used fibres. These are available in form of rovings (yarn) and woven fabric. Glass fibres, though not as stiff as Carbon and Kevlar, are mostly used due to their low cost.

Polymers, owing to ease in processing, good chemical resistance and low specific gravity, are most widely used matrix materials. Two types of polymer resins are available, thermoplastic and thermosetting. Thermoplastic resins softens on heating. Polyester resin, a type of thermoplastic is cheap, but it shrinks by about 4 to 8% (by volume) on curing, which is quite undesirable. Thermosetting polymers can not be reshaped by applying heat and pressure. Epoxy resins, a type of thermosetting resin, though expensive than polyester resins, has high adhesion to fibres and low shrinkage (about

3%).

In the work presented here, E-Glass fibres in woven fabric form and epoxy resins (thermosetting polymer) are used. Glass fibre-epoxy resin composite materials are most commonly used in aerospace industry, automobile industry, marine construction and sports goods. Typical products developed so far are boat hull, pipes & ducts, process & pressure vessels, pole vaults and secondary structures of aircrafts etc.

1.3 Fabrication of Glass Fibre-Polymer Composites

The fabrication of fibre composites consists of impregnation of the fibres with matrix material so as to wet the fibres completely. In case of thermosetting matrix composites, material formation accomplishes during moulding due to their curing characteristics (e.g. room temperature hardening epoxy resins). It cures fully, once hardener is added to the resin. These type of polymers can not be reshaped by applying heat once they are completely cured.

In case of thermosetting matrix composites, the first step is to form the thin laminas or preregs. This is accomplished by impregnating the fibres with resin and curing laminas partially. In the second step they are stacked to required thickness in a mould and cured under heat and pressure.

In other words, we can broadly classify the fabrication processes for polymeric composites as Wet forming processes and Dry forming processes using premixes or preregs (Lubin 1982).

Wet forming process consists of combining fibres and matrix together in a mould itself. Then curing is carried out at desired temperature and pressure. These include hand lay-up, filament winding, pultrusion and injection moulding.

In dry forming processes, forming and moulding operations are uncoupled. These

processes are divided into two steps,

- (i) Making prepregs and
- (ii) Producing components from prepregs.

Prepregs, a semi-cured product in thin sheet form, is made by impregnating fibre rovings or woven fabric and then semicured. Unidirectional prepregs are preferred, as they give maximum possible strength in the fibre axis direction. But it is more difficult to make unidirectional prepregs as compared to woven fabric prepregs. Once prepregs are available, the remaining process is rather simple. These prepregs are then used for product lay-up in a mould and final curing is carried out by applying required temperature and pressure (Molyneux 1983). Examples of these technique are hand lay up, matched die moulding and the bagging methods.

1.4 Wet Forming Processes

1.4.1 Hand lay-up

In this method, the constituent materials, fibres in chopped or woven form and thermosetting resins (e.g. epoxies and phenolic) are laid up or sprayed on a flat surface or a mould and is allowed to cure, without applying any pressure. The moulds are made up of wood, metal or plastics, having desired surface finish. Moulds for higher production volumes are generally made of hardened tool steel. The mould surfaces are polished, which determines the surface finish of the end product and its ejection from the mould after curing. Before lay up a gel coating is applied on this surface. Finally, fibre glass and resin mixture are applied onto the mould. Here the orientation of fibres can not be controlled very accurately. In spray lay up, chopped glass fibres and resin are simultaneously deposited on the mould surface through a specially made spray gun. Entrapped air is removed by using serrated rollers by compacting material against the mould surface. Then it is left for curing. In Dry forming process, preimpregnated tapes are used, other things remaining the same.

In hand lay-up, the fibre orientation being random, good use is not made of high fibre strength, except when continuous fibres are used. This technique needs minimum and low cost tooling. It gives only one finished surface. This process is very labour intensive, resulting in very low production volume, particularly with room temperature hardening resins. As the curing is carried out without application of high pressure and temperature, resulting composite has voids. The composite is weak, lacks strength and stiffness, necessitating thicker sections and consequently not exploiting the weight advantage to the maximum.

This method is particularly suitable for large or medium sized components of moderately low production cycles. Typical products made using this technique are boat and boat hulls, ducts, pools, air cooler bodies and tanks etc.

1.4.2 Filament Winding

In principle, this technique consists of wrapping required number of bands or layers of continuous fibres or rovings over a mandrel in a single machine operation. The mandrel decides the shape and size of the end product. The fibres may be impregnated in a resin bath (wet winding) before winding or preimpregnated (dry winding) or postimpregnated. After curing, generally at elevated temperatures, the mandrel is removed. Depending upon the winding angle, this process is classified as hoop (circumferential) winding, in which fibres are wound almost perpendicular to the mandrel axis and helical (longitudinal) winding, in which fibres are wound almost parallel to the mandrel axis. The winding angles and location of fibres are controlled with the aid of special machine to match the traversing speeds with mandrel rotation. The main advantage of this method is high production rates due to process automation. This method is best suited to manufacture surfaces of revolution such as pipes, tubes, cylinders and spheroids. Products of this process are rocket-motor case, missiles, helicopter blades for military applications and reinforced pipes, pressure vessels and wind turbine blades commercial applications.

1.4.3 Pultrusion

This process is analogous to extruding metal. Pultrusion is carried out through several variations, although the basic mechanism is the same. In the tunnel oven method, fibres rovings are impregnated in a resin bath and then drawn through a sizing bushing to remove entrapped air and excess resin and to impart the desired cross sectional shape. Then the composite passing through an oven is cured continuously in the heated chamber. The length of time, the composite requires for curing is governed by the length of heated chamber and the velocity at which it passes through the oven. After curing it is cut to required length with a cut-off saw.

The second variation of this technique is known as step-moulding pultrusion. Here fibres are drawn through the resin bath and then through a split female die. It is statically cured by externally heating the die. The input end is generally cooled to avoid the curing at the start of the die.

Pultrusion is primarily suitable for producing solid constant cross-section profiles like rods, tubes and beams with T and other cross sections. The main advantage of this process is its high speed of fabrication and high material utilization, about 95% compared to 75% that of hand lay up. The drawbacks are, limited flexibility of shapes and prone to inconsistent quality as resin sticks to the sizing bushings and the walls of the die.

1.4.4 Injection Moulding

This process is employed for the mass production of smaller sized objects, which are moderately stressed, with short-fibre reinforcement. Intricately shaped components of varying sizes, ranging from a small washer to fairly large components like moped bumper can be injection moulded. The raw material used for injection moulding is a compound of the resin and fibres in a palletized form and is prepared in a separate process. There are two methods of forming moulding compound - extruder compounding and trend

coating. In the former process, the chopped fibres and resin are fed directly into an extruder for mixing. The later consists of passing the rovings of fibres through an extruder die head so as to coat and impregnate fibres with resin which are subsequently cooled in a water bath and cut into required lengths. The process involves,

- (1) Softening thermoplastic or partially cured thermosetting plastic or resin containing reinforcing material and feelers by heating;
- (2) Forcing this softened plastic into a cooled matching mould through runners and spruces under high injection pressure;
- (3) Solidifying the plastic in the pressure clamped mould;
- (4) Mechanical ejection of solidified part from the mould.

It is worth mentioning, that even though the strength of short fibre composites is significantly lower than that of long fibre composites, this is the most widely used fabricating process of all composite fabricating processes.

1.5 Dry Forming Processes

1.5.1 Matched Die Moulding

In this method, the prepregs are stacked in a matching mould. The halves of these metallic moulds form the shape of the product. The curing cycle is 1 to 5 minutes, depending upon the part geometry and the type of resin, at preselected temperature. This method gives very good surface finish on either side. However very high cost of the dies, can only be justified when production volume is high.

1.5.2 Vacuum Bag Moulding

In this method, prepregs are laid up in a die as required and a film of cellophane, polyvinyl alcohol or nylon is placed over it. The ends are sealed with special sealing compounds. Vacuum is then drawn on the bag formed by this film, which removes excess air, feels voids and forces out excess resin. The excess resin is forced out in a suitably designed bleeder system. Vacuum connection is placed over this bleeder system, and

enough ports are provided to ensure uniform flow of resin. The bagging film is placed over the lay-up, bleeder system and vacuum connectors and is sealed to the mould plate. The entire system is transferred to an oven and full vacuum is applied for curing. This process is ideally suited for large size components which does not need high structural properties and which can be injection or press moulded

1.5.3 Pressure Bag Moulding

This involves placing a special bag usually made of rubber over the lay up and then using air or steam pressure to eliminate voids, force out entrapped air and drive out excess resin, which is collected in resin trap. Pressure upto 0.2 MPa (Schwartz, 1984) can be used depending upon the requirement. This requires a single die which avoids high cost of matching dies, but the entire set up is more expensive than vacuum bag or autoclave moulding, as it is combined with curing pressure system. Also it can be used only for the specific part for which it is designed.

1.5.4 Autoclave Moulding

Autoclave moulding is similar to vacuum bag moulding and is a modification of pressure bag moulding. The lay-up is subjected to greater pressure and denser parts are produced.

The bagged lay-up is cured in an autoclave by simultaneous application of heat and pressure. Most autoclaves use also vacuum assist in removal of trapped air. Vacuum is applied in the initial stages to remove entrapped air from inside of the bag and autoclave pressure in the range of 0.3 to 1.0 Mpa (3 to 10 atmosphere) is maintained during the entire curing. Compared with vacuum bag moulding, this process yields laminates with closer control of thickness and lower void contents.

A point to be noted for all bagging methods is that the bag has to be discarded after a single use.

1.6 Why A New Technique Is Needed?

In hand lay up, a open mould is used which substantially reduces the tooling cost. However, as curing is not carried out under pressure, void contents are high. This results in weaker composites, which means increased thickness of the sections, restricting weight advantage composites have over conventional materials. In vacuum bag technique, the effective pressure applied is 1 ata., which gives better composite than hand lay-up. But still voids are present in the composite as pressure is not sufficient. Pressure bag technique allows higher pressure to be applied but, it is not safe enough. So a question arises. Can we have a process which combines the advantages of some of the processes described earlier? Can we have a open mould, hand lay-up process in which fibre orientation can be controlled, curing carried out at elevated temperature and high pressure? Which can make use of either prepregs or wet lay-up? And still be economical and safe?

A new technique named as **Balloon Compression Moulding** is developed in the present work to overcome some of the limitations of the conventional composite fabricating technique.

1.7 Layout of The Present Work

Current chapter has presented an introduction to the polymer composite fabrication techniques. Chapter 2 presents the basic principle methodology, design of required tooling, and other details of Balloon Compression Moulding. The characterization of the products fabricated using this technique and discussion is presented in chapter 3. Chapter 4 concludes the work and suggests further work for enhancement of this technique.

CHAPTER 2

BALLOON COMPRESSION MOULDING

2.1 Background

Although polymer composites offer properties like high strength, light weight (high specific strength), corrosion resistance and various others, one wonders why these materials are not being used extensively in engineering applications? Why their use is mainly confined to military and aerospace applications? The factors hindering widespread application of composite materials in engineering applications are high cost of raw materials, complex and expensive tooling and high rejection due to inconsistency in quality, in all making composite material considerably expensive than metals. Aerospace and military applications demand low weight and high strength of a material

without too much consideration of costs. But for engineering application, high material cost can not be justified in this highly competitive market. A need is felt to reduce composite material cost by developing a cost effective fabricating process.

2.2 Objectives

After having a brief review of the existing fabricating techniques, a need is felt to develop a technique of composite material fabrication which should have the following features. Tooling design should be simple, reducing the tooling cost. The tooling can be made using easily available materials and manufacturing facilities, resulting in lower cost per item produced. The technique should be able to use fiber reinforcement in any form - rovings, woven fabric or preimpregnated (prepreg) tapes. The technique should be able to produce components having, double curvature or complex shapes.

2.3 Components Developed by Balloon Compression Moulding

Balloon compression moulding is a general composite fabricating technique which can be applied to variety of products. Separate tooling will be required for each product. In this work, tooling was developed for three different products. These products are;

- 1 Circular ring (Fig. 2.1),
- 2 Convex-concave surfaced ring (Fig. 2.2) and
- 3 Circular ring with concave cross section, resembling a cycle rim (Fig. 2.3).

The circular ring was made at the beginning so as to have confidence in the entire process. The convex-concave surfaced ring was developed next. This illustrated that the balloon applies equal pressure everywhere on the laminas and that laminas take the mould shape. Circular ring with concave cross-section again proved that complex shapes can be produced using this method.

2.4 Principle of Balloon Compression Moulding

In this technique a donut shaped balloon is used to apply normal pressure on the stacked laminas in an open mould. The basic idea of this process is to lay-up the fibres, impregnate in the mould and apply normal pressure by inflating donut shaped balloon for carrying out curing. When the balloon inflates, the laminas are pressed against the mould walls. This makes excess resin to bleed out.

Essentially it consists of a single mould plate and a donut shaped balloon as shown in the schematic diagram (Fig. 2.4). Single mould plate geometry corresponds to that of the product being fabricated. Fibres are impregnated with resin and are laid-up on the single mould surface, and the donut shaped balloon put in place. The top plate and bottom plate are clamped together and balloon is inflated, which applies normal pressure on the laminas.

In hand lay-up technique, curing is not carried out under pressure. This results in not so dense and resin rich products with high void content, which is not the case with balloon compression moulding. Curing can be carried out at elevated temperatures if need be, to get required properties in this technique.

Axi-symmetric components like pullies, flywheels, gears can be easily moulded, without incurring heavily in tooling cost. Even if these components have some negative curvature, they can be successfully moulded. The very best example that of a radial cam, which has complex double curvature, can be easily moulded.

2.5 Lab Scale Set-up

2.5.1 For Circular Ring

Main components of the lab scale set-up are; Mould plate, donut shaped balloon, see through top plate, bottom support plate and core (Fig. 2.5).

Bottom support plate (1) is circular mild steel plate. Six bolts of M 10 x 60 mm (2) are welded to this bottom plate on 200 mm pitch circle diameter.

The mould plate (3) is made up of aluminium and is finely machined and polished. The geometry of mould plate corresponds to that of the product being fabricated. If the product design allows, a draft of about 1.5° is recommended to facilitate easy removal of the product from the mould after curing. If the product height (width of the ring in this case) is small, draft may not be required. In case the height of product is more and product design does not allow draft, an eject mechanism can be provided or a split type mould designed. Mould plate sits on the bottom support plate, the bolts passing through the holes on pitch circle diameter.

The core (4) is made of mild steel and rests on the bottom support plate with a cap screw (5). The donut shaped rubber balloon (6), sits in the cavity between the aluminum mould and the core. The balloon when expanded fills this cavity and applies normal pressure on the laminas. The volume of the balloon, in inflated condition is slightly more than the volume of the cavity (between the mould plate and the core).

The top plate (7) is made of transparent plexiglass, which also has six holes on pitch circle diameter for the bolts, to pass through them. It also has provision for the valve (8) of balloon to come out.

The entire assembly after lay-up is fastened with nuts (9) and the balloon inflated to the required pressure. The laminas(12) are laid up in the mould. Nitrogen at high pressure was used to inflate the balloon. The pressure was continuously measured with the help of an pressure gauge.

The entire assembly rests on a stand (10), made of mild steel channels (30 x 30 x 6 mm), welded together. The assembly is bolted to the stand with the help of four allen head screws. The assembly, without the stand can be put in a oven for curing with heat if required.

Figures 2.6 and 2.7 show the photographs of the experimental set-up. Figure 2.8 shows the main components of the set-up.

It is worth noting that, top plate and bottom support plate are provided with a 4 x 4 mm deep undercut, for the following reasons. The balloon is made of rubber. The balloon, when inflated, has minimum radius of curvature of about 2 mm. Without an undercut on the top and bottom, the top and bottom portion of about 2.5 mm of the product in axial direction is cured without application of any pressure. With this undercut, the balloon's radius of curvature has been shifted 4 mm away from the moulding surface on either side in axial direction.

The epoxy resin used in the work cures in 24 hours at room temperature. An infra-red lamp was used to maintain a mould temperature of about 35~40°C.

2.5.2 For Convex-Concave Surfaced Ring

For a convex-concave surfaced ring, the mould plate is made of aluminium and has a contour (Fig. 2.9) corresponding to the geometry of the component being moulded. The core (Fig. 2.10) is also contoured, so as to allow the rubber balloon to take its shape. Photograph of the core and mould plate is shown in Figure 2.11. The other components remain the same as those used for circular ring.

2.5.3 For Circular Ring with Curved Cross-section

For a circular ring with concave cross-section, a two-piece mould plate (Fig. 2.12 and Fig. 2.13) of mild steel is manufactured. The ring, having concave cross-section, physically can not come out of a single piece mould plate, necessitating a two-piece mould. Two halves of this mould were joined together with two M 8 bolts and then machined, to get desired accuracy of alignment. After the component has been cured, the two halves are separated, by unscrewing the bolts and taking the casting out. The other components remain the same as those used for circular ring.

2.6 Moulding

2.6.1 Preparation for Moulding

The entire set-up is cleaned with acetone to remove any resin left over. The mould is polished with a fine emery paper before moulding. A coat of polyvinyl alcohol is then applied on mould surface. After two hours one more coat is applied. This acts as mould release agent and assists easy removal of the component after curing. The bottom support plate, the top plate and the balloon are also coated with polyvinyl alcohol.

2.6.2 Moulding

Woven glass fabrics are cut to the required width. The circumferential length of fibres is made up of four pieces, to allow sliding of woven glass fabric during outward radial motion during compression. These fibres are impregnated with epoxy resin. The thickness is built by stacking required number of layers (usually 16 layers to get 2.8~3 mm thickness). A thin tape of teflon is placed on the inner surface of the laminas, avoiding direct contact of epoxy and rubber, and it gives better surface finish on the inner side. The donut shaped balloon is placed inside the cavity and the mould is closed. The donut shaped balloon is inflated to a pressure of 0.45 Mpa to 0.65 Mpa (4.5 to 6.5 ata), depending upon fiber volume fraction requirements. The excess epoxy, which comes out settles in the undercuts made in the top and bottom support plates. The type of epoxy resin used (LY 556 and HY 951 of Hindustan Ciba-Geigy, Mumbai) has 24 hours curing cycle. The epoxy starts getting into jelly stage after about 45~50 minutes. Therefore lay-up is must be applied quickly so that pressure is applied within 30 minutes epoxy is mixed with hardener. Because the room temperature was below 20°C at the time of most of the fabrications, the entire set-up was heated to 35-40°C with the help of an infra-red lamp. Pressure in the balloon was maintained for next 24 hours for curing. After curing the balloon is deflated and the mould plate is removed. The product is taken out from the mould plate. Slight finishing, like cutting is required to maintain the width of products.

We can also use prepreg tapes for making the product. This will facilitate easy and clean moulding. But the difficulty experienced was the curing temperature. The unidirectional prepreg made at ESA lab, IIT Kanpur uses epoxy resin (LY 556 and HT 973 of Hindustan Ciba-Geigy, Mumbai) with fiber glass rovings. This epoxy has a curing cycle of four hours at 130°C and pressure of 1 Mpa. The donut shaped rubber balloon used in this study is made of an ordinary racing cycle tube (1 1/4 inch), by joining the two ends together. The joint does not withstand temperature beyond 50°C, because it starts leaking at elevated temperature. The reliability of the joint even at room is not high because the joint is designed for pressure upto 0.2 Mpa, whereas pressure of 0.5 to 0.7 Mpa is preferred. Furthermore the wall of the rubber balloon is quite thick, which does not take mould shape very easily. However, this is not the limitation of this process. A thin seamless balloon of required strength at elevated temperature (upto 150°C) and pressure can be developed to overcome these difficulties.

2.7 Closure

A new technique is developed which overcomes some of the limitations of the existing composite fabricating techniques. Three different products with varying degree of complexity of geometry are fabricated.

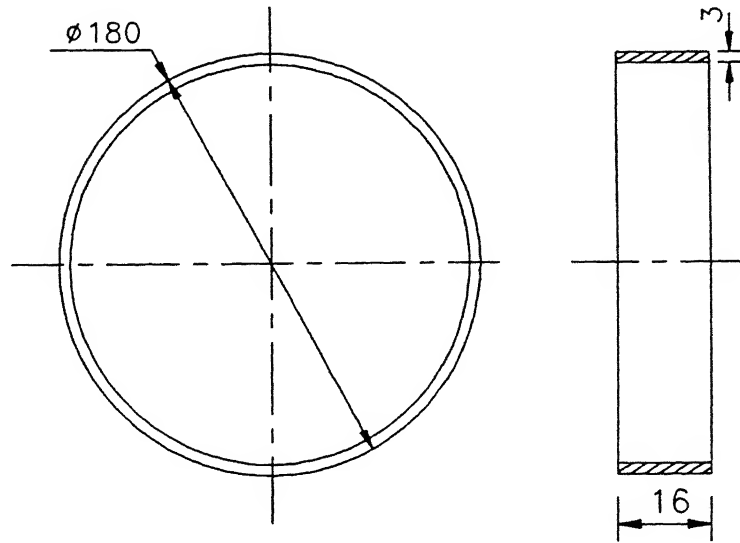


Figure 2.1 Drawing and Photograph of Circular Ring

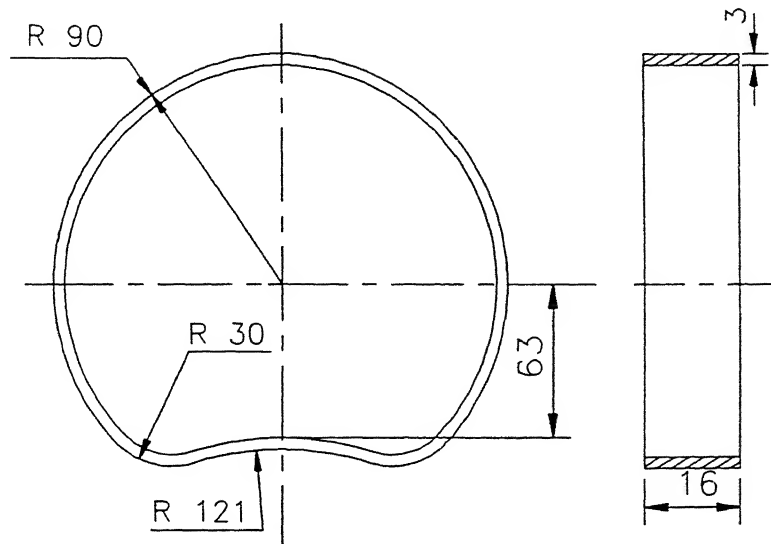


Figure 2.2 Drawing and Photograph of Convex-concave Surfaced Ring

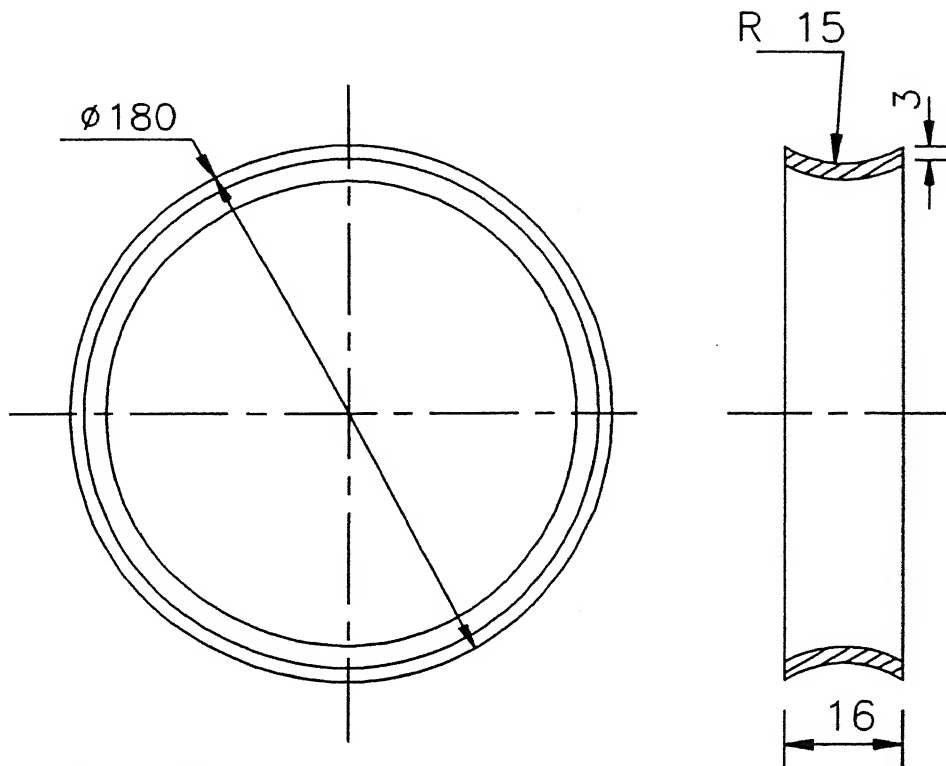


Figure 2.3 Drawing and Photograph of Circular Ring With Concave cross section

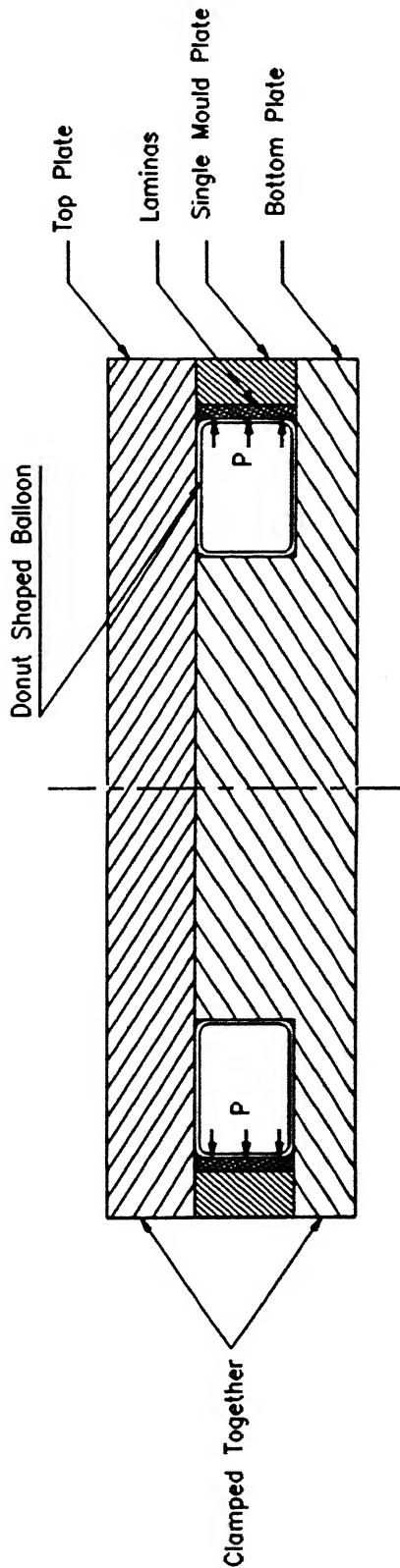


Figure 2.4 Principle of Balloon Compression Moulding

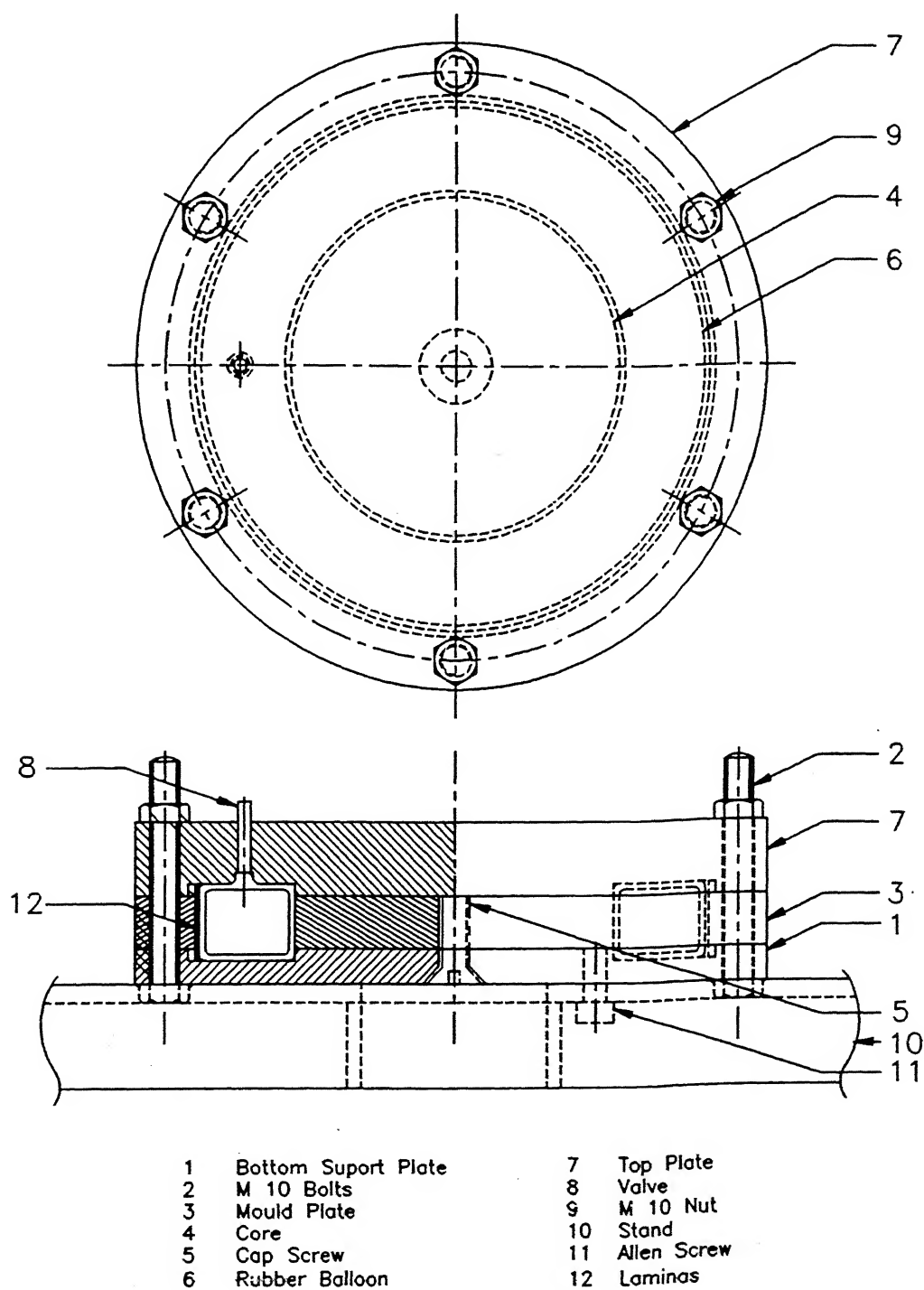


Figure 2.5 Half Sectional View
of Lab Scale Set-up

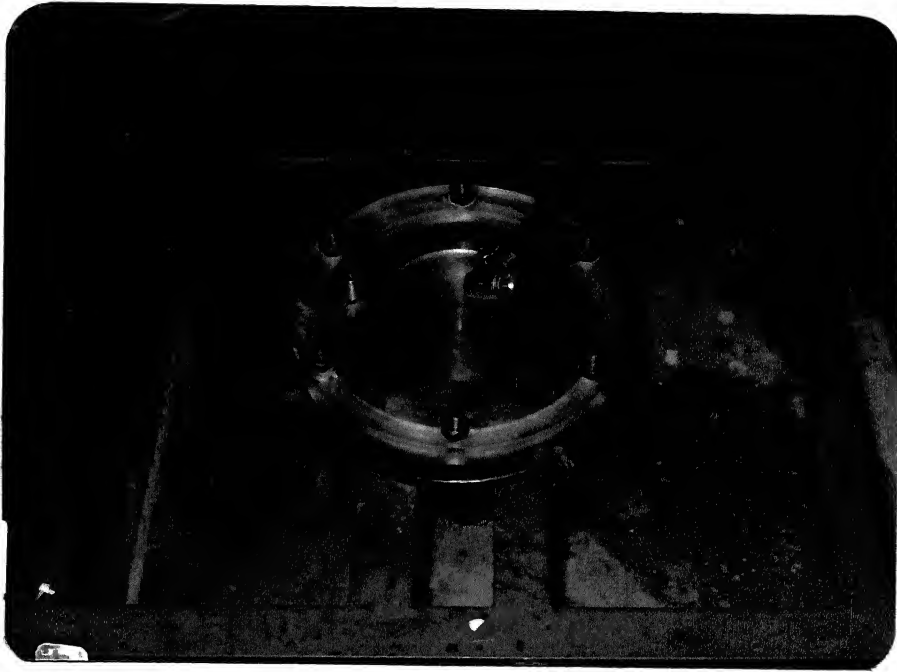


Figure 2.6 Photograph of the Experimental Set-up

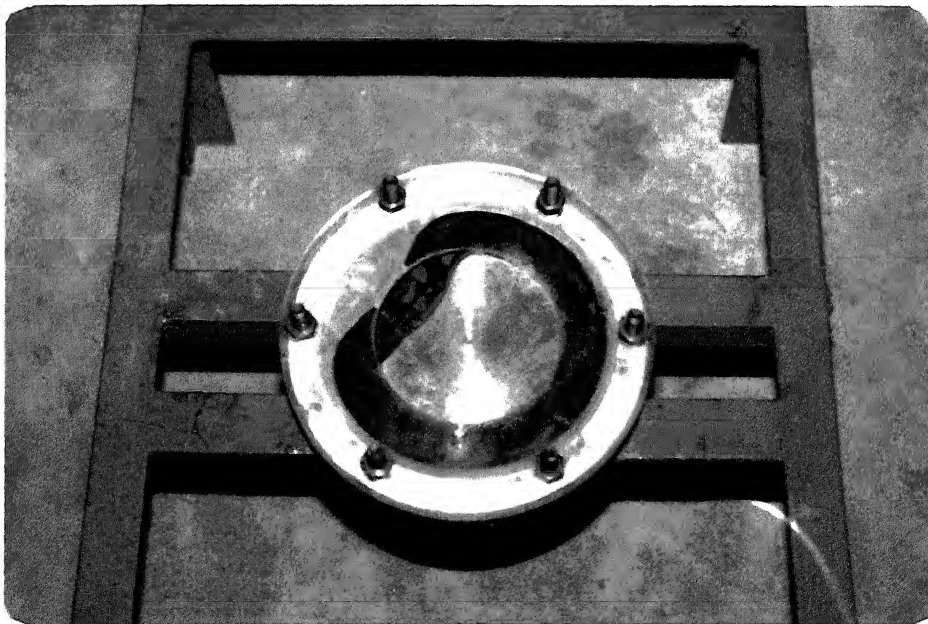


Figure 2.7 Close-up of the Experimental Set-up



Figure 2.8 Photograph Showing the Components of the Set-up

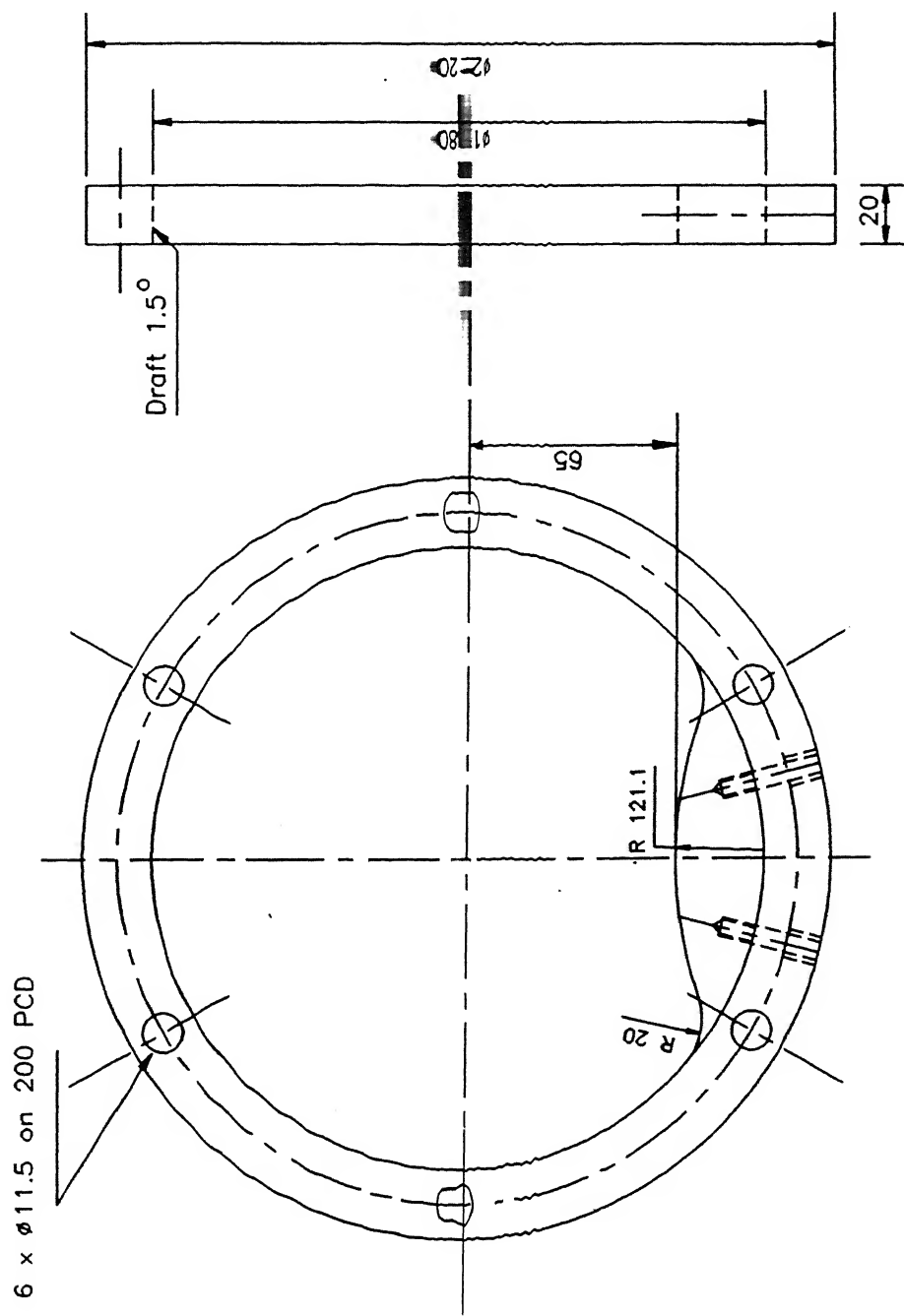


Figure 2.9 Mould Plate for Convex
—concave Surfaced Ring

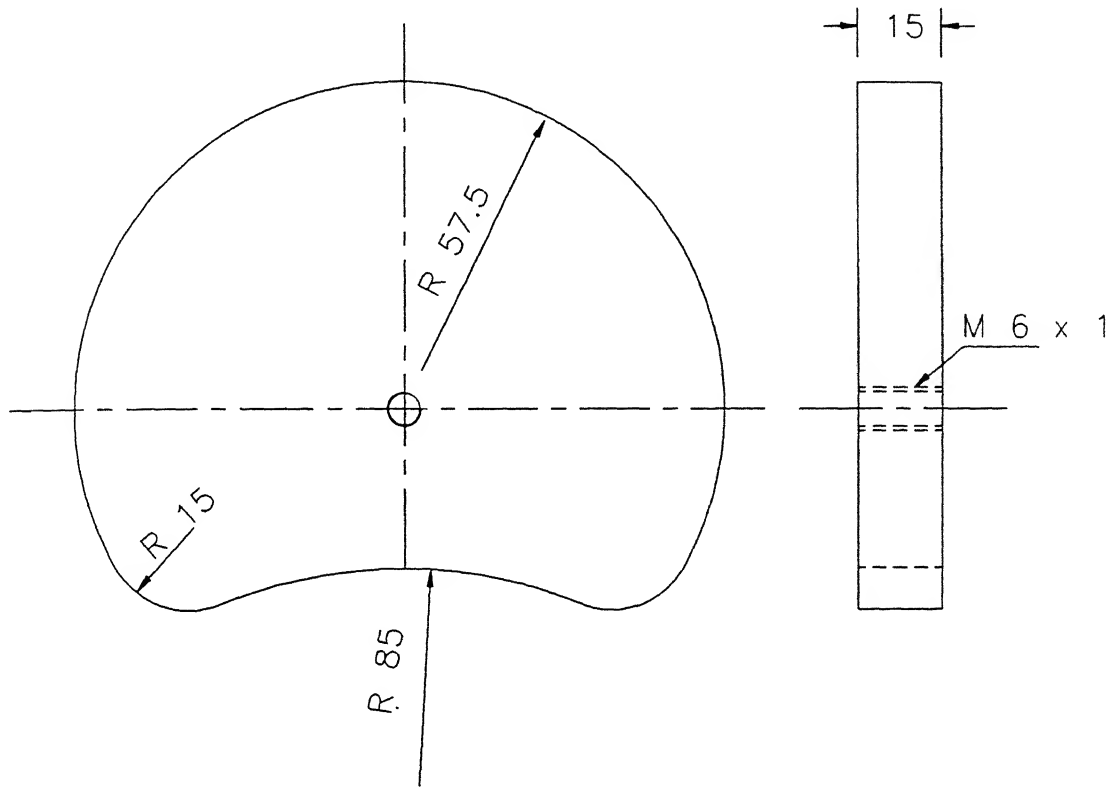


Figure 2.10 Core For Convex-concave Surfaced Ring

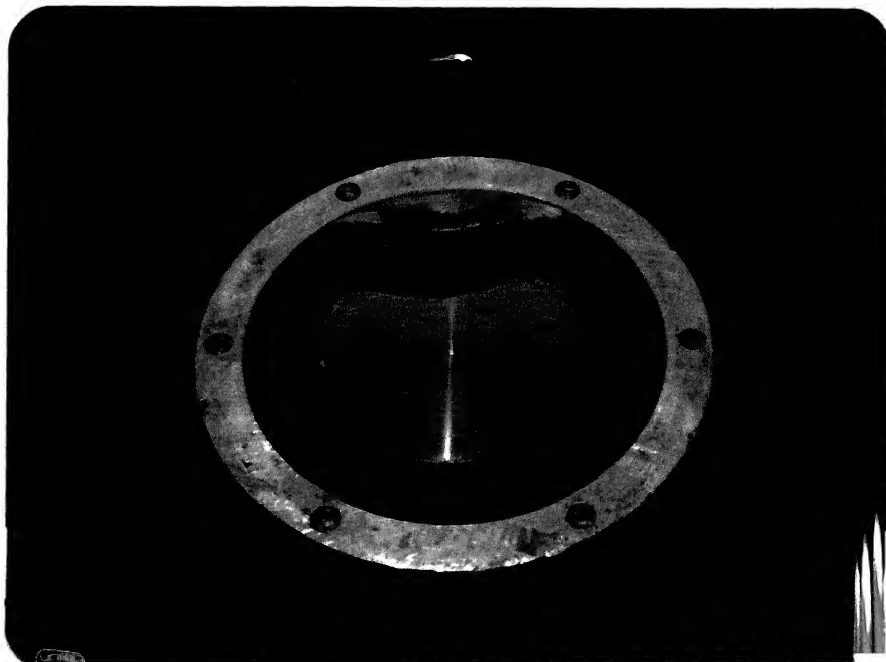


Figure 2.11 Photograph Showing Core and Mould Plate
Convex-concave Surfaced Ring

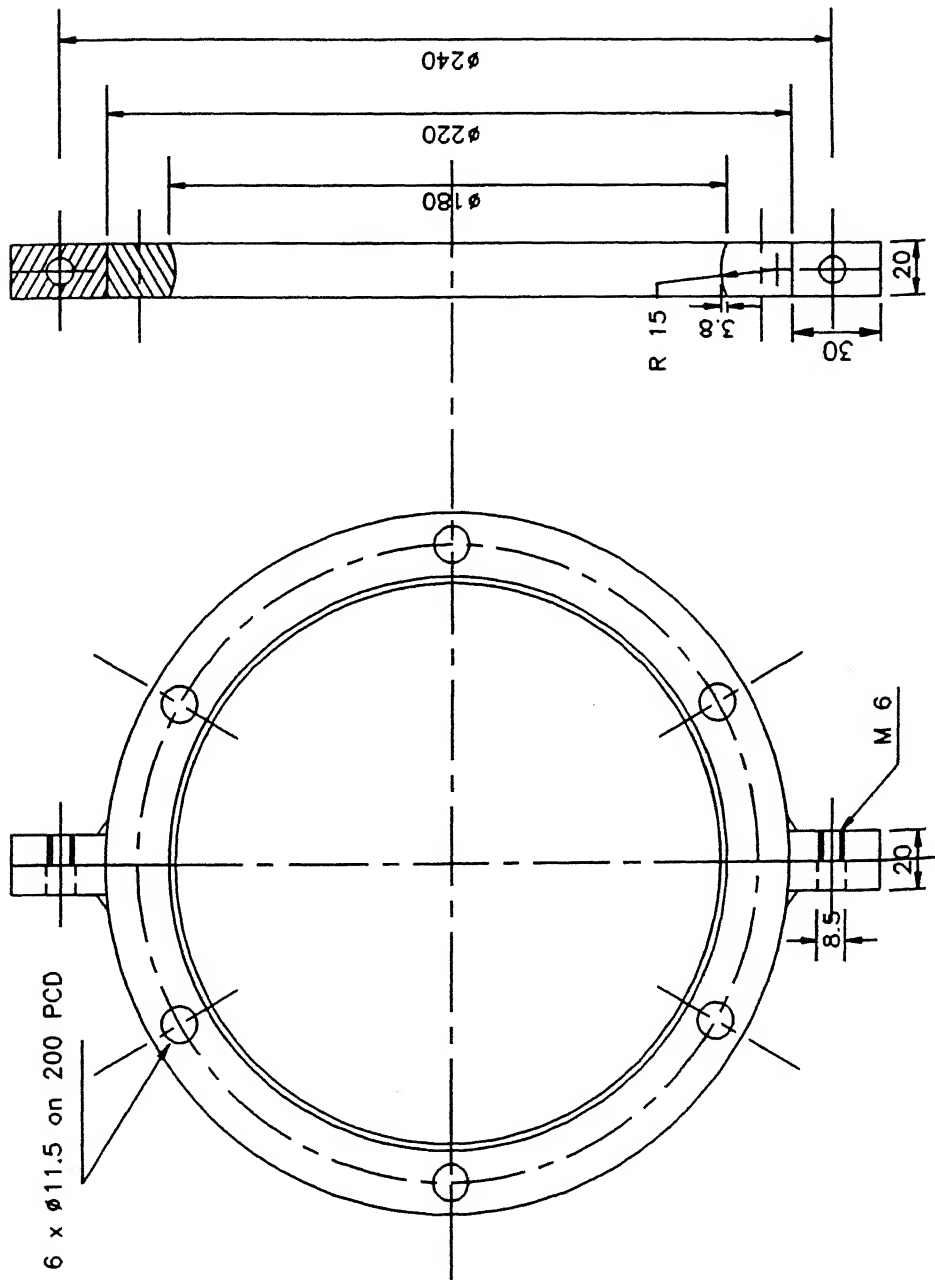


Figure 2.12 Two Piece Mould for Circular Ring with Curved Cross-section

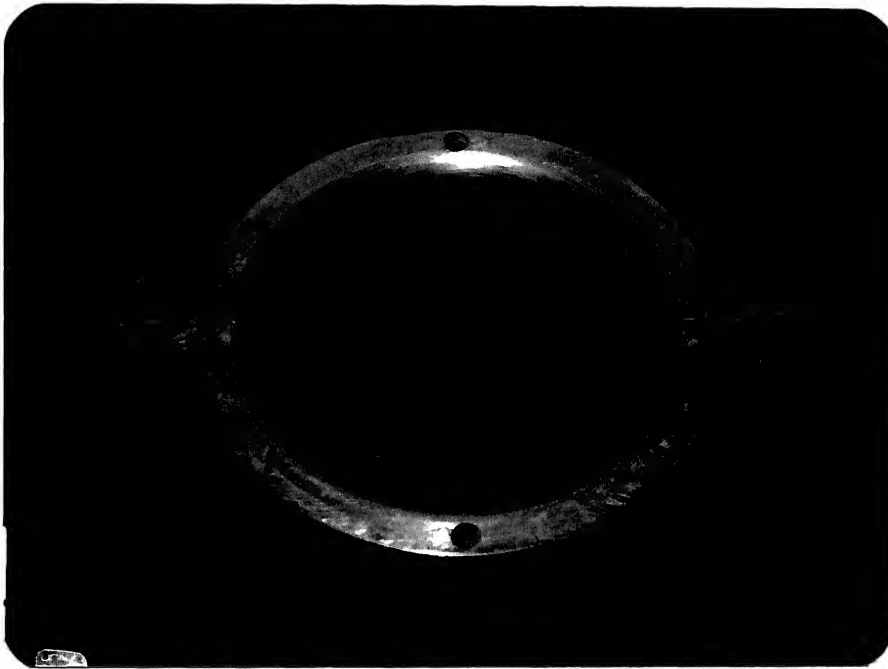


Figure 2.13 Photograph of the Two-Piece Mould for Circular Ring with Concave Cross-section

CHAPTER 3

PRODUCT CHARACTERIZATION AND DISCUSSION

3.1 Introduction

For a designer, the mechanical properties of the composite is the most important data required to arrive at the physical dimensions of the end product. Designer will determine the physical dimensions of the products in such a way that the stresses in the product does not cause failure of the component. The mechanical properties of polymer composites can be estimated fairly closely, by using the rule of mixture (Agrawal 1990).

But an experimental investigation is necessary to ascertain the validity of the theoretical determination of the properties.

Fabrication process has the most significant influence on the properties of polymer composite materials. The curing cycle in particular imparts the mechanical properties on the composite. The other significant parameters which affect the end mechanical properties is the tooling itself. The curing pressure controls the resin bleed-out and ultimately the fibre volume fraction. The curing temperature determines the duration of cure cycle and imparts toughness properties. Curing temperature for a particular type of epoxy resin is taken from the manufacture's catalogue.

3.2 Objectives of Characterization

In order to evaluate the properties of the products fabricated through the Balloon Compression Moulding and hence the usefulness of the process, characterization was carried out. The tests were carried out for the following;

- 1 Determine the physical dimensions of the product. This is to check the uniformity in thickness and circularity, and hence process capability.
- 2 Determine the fibre volume fraction of the products. It is well known, that the final composite properties are function of the volume of the fibres and matrix materials and their respective properties.
- 3 Determine the mechanical properties such as ultimate flexural strength and modulus of elasticity. To compare the values of strength and elastic constant, flat laminates of the same constituent materials from same batch was prepared with identical curing conditions. Using standard test method (ASTM 3039) these laminates were characterized.
- 4 The polished specimen of the products were analyzed under a Scanning Electron Microscope. The aim was to study the microstructure of the composite, presence of voids and debonding and delamination if any. The specimen fractured in flexure test were also analyzed to find the mode of failure.

3.3 Properties of The Constituent Materials

Before we move on to the characterization aspect, the properties of the constituent materials are presented.

3.3.1 Glass-fibre Fabric

E-class glass fibres in woven form supplied by Ceat Ltd, Mumbai are used. Specifications of these fibres are given in Table 3.1.

Table 3.1 Specifications of Glass-fibre fabric

Fabric form	Untwisted, double weave
Nominal thickness	0.44 mm
Area density	220 gm/m ²
Count	50-50, Bi-directional

3.3.2 Matrix Material

Matrix material is Epoxy Resin LY 556 and Hardener HY 951 supplied by Hindustan Ciba-Geigy Ltd, Mumbai.

The term epoxide refers to a chemical group consisting of an oxygen atom bonded with two carbon atoms already united in some way. Epoxies are obtained by epoxidation which is the introduction of three membered ring into an organic compound. The basic characteristics of epoxy resins are; easy cure, low shrinkage and high adhesive strength.

Formulation of Epoxy Resin Mixture

The impregnating solution used has following composition at room temperature.

Araldite LY 556	100 parts by weight
Hardener HY 951	10 parts by weight

Araldite LY 556 is a liquid, unmodified bisphenol-A epoxy resin. The properties of LY 556 and HY 951 are given in Table 3.2 and 3.3 (Ciba 1994) respectively.

Table 3.2 Properties of Araldite LY 556

Viscosity at 25°C	9000-12000 Mpa s
Epoxy content	5.2-5.45 equiv/kg
Density at 25°C	1.1-1.2 gm/cm ³
Flash point	> 200°C
Storage life	5 years

Table 3.3 Properties of HY 951

Viscosity at 25°C	5000-8000 Mpa s
Density at 25°C	1.1-1.2 gm/cm ³
Pot life after mixing with LY 556	0.5 - 1 hours

This resin and hardener mixture has a very low pot life of about 45 ~ 50 minutes at 25°C. The curing cycle at this temperature is;

Gelation starts	45 ~ 50 minutes
80% curing	8 hours
100% curing	24 hours.

The curing time is a function of temperature. At a temperature of 150° to 175°C, the curing time is 1.5 ~ 2 minutes. However all experiments were carried out at room temperature.

This particular Epoxy-Hardener combination gives very low cure shrinkage, dimensional stability and small internal stresses in a product. Moreover, the products possess good mechanical and electrical properties. They are resistant to weathering and humidity. The products are not affected by most of the chemicals used in industries and have excellent water resistance. They do not cause corrosion and have low thermal conductivity and a low burning rate. Products may be coloured with Araldite colouring paste and are easy to machine. (Ciba 1992).

3.4 Characterization of Circular Ring

Circular rings (Fig. 2.1) were fabricated using Balloon Compression Moulding.

3.4.1 Dimensional Stability

The diameter, thickness and width of circular ring (Fig. 2.1) were measured using a vernier calliper. For measuring circularity, the ring was placed on a V-block. A dial indicator was set to zero, then the ring was slowly rotated and readings noted down. Table 3.4 provides the details of the dimensions achieved.

Table 3.4 Dimensions of Circular Ring

No.	Specimen No.	Avg. Outside dia. (mm)	Avg. Inside dia. (mm)	Avg. Thickness (mm)	Avg. Width (mm)	Circularity (mm)	Thickness variation (mm)
1	R1*	179.5	173.2	3.2	16.3	0.8	0.5
2	R2*	179.6	173.0	3.2	16.2	1.0	0.43
3	R3	178.9	173.46	2.72	15.44	1.3	0.28
4	R4	179.6	174.0	2.95	16.8	1.0	0.35
5	R5	180.0	174.2	3.0	16.75	0.5	0.4

* Pressure could not be applied for complete curing as the balloon leaked during the experiment and the pressure was dropped to zero.

The results show that variation of circularity is within 1.3 mm, which is quite reasonable. However variation in thickness is quite large (0.5 mm). The main reason for this variation is non uniform curing pressure over the laminas. The rubber balloon is made from an ordinary cycle tube, cut to length and joined. The overlapped length of this balloon is about 35~40 mm (minimum required for joining operation), usually upto double the thickness of balloon. It is evident that at the joint the balloon would not inflate to the extent it will at other areas of balloon. Furthermore there are other some reasons also;

- (i) The hand lay-up was crude, with overlap of glass-fibre fabric at certain places;
- (ii) Resin was applied with a brush and was quite uneven and
- (iii) The viscosity of resin is on higher side, which does not allow full resin bleed-out. Further, the pot life of epoxy is so short that, by the time the assembly is completed and pressure applied, the epoxy mixture is close to gelation and there is not enough time for epoxy bleed-out.

3.4.2 Fibre Volume Fraction Determination

The fibre volume fraction is another important property, which determines the contribution of fibres and matrix materials to the properties of composite material. The relative proportions can be given as the weight fractions or the volume fractions as follows (Agrawal 1990):

Weight fraction of fibres (W_f) and matrix (W_m) is given by,

$$W_f = \frac{w_f}{w_c} \quad (3.1)$$

$$W_m = \frac{w_m}{w_c} \quad (3.2)$$

where, w_f = weight of fibre

w_m = weight of matrix

w_c = weight of composite material

To find volume fractions from weight fractions, the density ρ_c of the composite material must be obtained. The conversion between the weight fraction and volume fractions can be obtained by the following expression:

$$V_f = \frac{\rho_c}{\rho_f} W_f \quad (3.3)$$

$$V_m = \frac{\rho_c}{\rho_m} W_m \quad (3.4)$$

where, V_f and V_m are fibre and matrix volume fractions while ρ_c , ρ_f and ρ_m are density of composite, fibre and matrix respectively.

The burn test is adopted, in this study, to determine fibre volume fraction. In this method, the specimen of laminate (approximately of area 20 mm x 20 mm) is weighed in air (w_{ca}) on a weighing machine capable of measuring weight with the accuracy of 0.0001 gm. To determine the density of the composite, it is necessary to find the weight of the same specimen in water (w_{cw}). This is obtained with the help of specially designed stand (Kulkarni 1995). Thus, the density of composite can be obtained as follows:

$$\rho_c = \frac{w_{ca}}{(w_{ca} - w_{cw})} \quad (3.5)$$

The specimen is then placed in a furnace at a temperature of about 750 to 800°C for 2-3 hours till the matrix is burnt out. The residue is cooled and reweighed to obtain weight of fibres, w_f . The fibre weight fraction and hence volume fraction can be determined as described earlier. The test is quite a simple test, but is very effective.

The curing pressure determines the fibre volume fraction, by controlling the resin bleed out. The results of fibre volume fraction are presented in Table 3.5

Table 3.5 Fibre Volume Fraction

Sr. No.	Specimen No.	Curing Pressure (kg/cm ²)	fibre volume fraction (%)
1	R3	5.0	47.53
2	R4	4.5	32.32
3	R5	4.5	39.69

As listed in Table 3.5, the fibre volume fraction of the circular ring fabricated balloon compression moulding is in the range of 32 ~ 47%.

3.4.3 Determination of Flexural Strength and Modulus

The circular ring was loaded in diametral compression in an Inston testing machine in displacement control mode. The load and corresponding displacement were recorded.

For thin isotropic circular ring ($d/h > 20$), under diametral compression, close form analytical results are available using liner elasticity approach (Roark 1966). The loading diagram is shown in Fig. 3.1.

Bending moment (M), for ring having its thickness much smaller than diameter is given by,

$$M = PR(0.3183 - 0.5Z) \quad (3.6)$$

It is worth noting that circular rings fabricated in this study have diameter to thickness ratio greater than 55.

Maximum bending moment (at point A in Fig. 3.1) is

$$M_{\max} = 0.3183 P_{\max} R \quad (3.7)$$

where

P Load supported by ring and

R Mean radius of ring.

the flexure strength (σ) is obtained as.

$$\sigma = \frac{M \frac{h}{2}}{I} \quad (3.8)$$

where

h Ring thickness and

I Moment of inertia of cross-section,

The deflection (δ) is given by,

$$\delta = \frac{0.149PR^3}{EI} \quad (3.9)$$

where

δ Deflection and

E Youngs modulus of elasticity.

For the measured collapse load P_{\max} , flexural strength σ_{ult} can be determined through Eqs. 3.6 and 3.7. Modulus E is determined using Eqn. 3.8 from the linear portion of load-deflection curve.

The ring is symmetric, made up from a 0/90, 50-50, bi-directional woven glass cloth. So the material is orthotropic i.e., has identical properties in hoop (θ) and axial (Z) direction. Thus the circular ring is quasi-isotropic and hence the formulas for the flexural strength and modulus of elasticity for isotropic material are used to establish the flexural strength and modulus of the ring.

The photograph of the deformed circular ring during the experiment is shown in Fig. 3.3. The load-displacement curve, shown in Fig. 3.3, provides the ultimate load supported by the circular ring.

Table 3.6 Flexural Strength and Modulus of Circular Ring

Sr. No.	Specimen Number	Fibre Volume fraction (%)	P_{ult} (N)	δ_{max} (mm)	σ_{ult} (Mpa)	E (Gpa)
1	R3	47.53	172.5	52.5	257.53	18.16
2	R4	32.32	200.5	40.2	224.27	20.55
3	R5	39.69	237.5	55.5	266.28	23.97

The average flexural strength of the circular ring is 249.36 Mpa and modulus of elasticity is 20.89 Gpa. To compare the strength and modulus values, plain laminates were tested and will be discussed in subsequent section.

3.4.4 Tensile Strength and modulus of Plain Laminate

A tensile test was carried out on a plain laminate, to have more reasonable assessment of strength and modulus of the circular ring. A 16 layered laminate was prepared through conventional method using a hydraulic press. Glass-fibre fabric and identical resin formulation from the same batch were used and the laminate cured under identical conditions.

The American Society of Testing Metals (ASTM) standard test method for properties of fibre-resin composites has the designation D 3039 (Reapproved 1982). The geometry of the specimen and end tabs with their dimensions are shown in Fig.

The test has been conducted in an MTS testing machine of 10 ton capacity with a loading rate of 0.5 mm/m in the longitudinal direction in tensile mode. The ultimate load applied on the specimen is noted from the load versus displacement curve and tensile strength has been determined. The load-displacement curve is shown in Fig.

The initial slope of the load-displacement curve is found to obtain tensile modulus of the specimen. Table 3.7 presents the values of tensile strength, tensile modulus for various specimens along with their fibre volume fractions.

Table 3.7 Tensile Strength of Plain Laminate

Sr. No.	Specimen No.	Fibre Volume Fraction (%)	Tensile Strength (Mpa)	Modulus of Elasticity (Gpa)
1	L1	55.62	330.5	22.50
2	L2	55.62	326.3	21.98

The failure mode observed was interface matrix shear failure.

3.5 Characterization of Convex-Concave Surfaced Ring

This was first of the complex shapes produced using balloon compression moulding. The characterization was carried out to determine the quality of the lay-up, curing and drapability of the lay-up (i.e. to get cast in the mould shape).

3.5.1 Visual Observations

The visual observations showed, the surface to be well cured, with good surface finish on either side. The form is well taken except at the change of radius. The corner radii of about 20 mm was filled well, but the region is resin rich one. The mould, being a two piece mould (Fig. 2.7), the change in radius was not uniform. But the larger radius (90 mm) is very well taken, without any resin rich regions.

3.5.2 Dimensional Stability

The dimensional stability analysis showed a thickness variation of about 0.8 mm on either side. The thickness was on higher side in the resin rich regions. The reasons for this variation are identical with those for circular ring.

3.5.3 Determination of Fibre Volume Fraction

The average fibre volume fraction obtained was 49%. Again the possible reasons for lower fibre volume fraction are, lower curing pressure and short shelf life of epoxy mixture.

3.6 Scanning Electron Micrography

Polished surfaces of the circular ring were analyzed under Scanning Electron Microscope JSM 840. The specimens were cut to size and then finely polished. Specimens from both in hoop direction and Z direction were prepared. The primary aim of this microscopy was;

- * To study compaction of the lay-up,
- * To check presence of voids and their size and
- * To study the fibre-epoxy interface.

Voids were observed but, their size and number were fairly minimal, suggesting of proper lay-up and curing. Specimens were also prepared from the fractured circular ring (i.e the one tested in diametral compression). In a fractured specimen, delamination and fibre debonding was observed.

3.6.1 Micrographs of Virgin Specimen

The photographs of micrographs of virgin specimen in Z plane are shown in Fig. 3.6. Figures 3.6 (a) and (b) shows the microstructure of specimen R1 at magnification of 100 and 250 X respectively. The fibres are well impregnated. The white spots observed are

silicon particles. One or two voids are also seen.

Figures 3.6 (c), (d) and (e) shows microstructure specimen R2 at higher magnification. Figure 3.6 shows that fibres well impregnated with resin. An impurity is seen in the centre of the specimen. Figure 3.6 (d) shows fibre at high magnification of 5000 X. Proper wetting of fibres is visible. Figure 3.6 (e) shows presence of a void of the size < 5 micrometers, smaller than fibre diameter. Hardly 2-3 voids were observed in 50 mm^2 area scanned.

3.6.2 Micrographs of Fractured Specimen

Figure 3.7 shows the micrographs of the fractured specimens. Specimens were cut from the intact area (at point B in figure 3.1). Figure 3.7 (a) is for Specimen R3 after it had failed in flexure test. The fibre debonding is clearly visible. It is even more visible at higher magnification of 5000 X as seen in figure 3.7 (b)

Figure 3.7 (c) is cut area near A in Fig. 3.1 for the specimen R4 after the flexure test. It shows breakage of the fibres.

The white spots visible on the surface were first thought to be voids. But later an analysis showed, these to be silicon particles. It meant that the emery paper used for polishing has left tiny silicon particles on the polished surface.

3.6.3 Micrographs of Convex-concave surfaced ring

The microscopic analysis show similar results as obtained in case of circular ring (Fig. 3.8). Figures 3.8 (a), (b), (c) and (d) shows the microstructure of convex-concave surfaced ring at various magnifications from 180 to 5000 X. The fibres are well impregnated and product is well cured, as seen from one or two voids only. This is more clearly visible at higher magnification.

3.7 Discussion

The variation in thickness was 0.5 mm and circularity was within 1.3 mm. Some of the reasons were cited in section 3.4.1. The remedies to overcome the above mentioned shortcoming are,

- 1 Use of a thin walled, one piece balloon.
- 2 Use of preimpregnated tapes.
- 3 Use better lay-up procedure like automated lay-up etc.

The fibre volume fraction obtained was 47.53%, which is only marginally lower than fibre volume fraction of a plain laminate. For a woven fabric composite, fibre volume fraction of 50~55% is termed as optimum. The reasons for obtaining lower fibre volume fraction in this study are low pressure and short pot life of resin mixture. It seems that higher pressure in the range of 0.8 Mpa is required to achieve fibre volume fraction of about 55~60%. The shorter pot life of resin mixture (about 45~50 minutes) means, the pressure must be applied before the gelation starts, otherwise excess epoxy will not bleed out.

Average flexural strength obtained was 249.36 Mpa and modulus of elasticity 20.89 Gpa. These are for average fibre volume fraction of 39.85%. The average tensile strength and modulus of plain laminate obtained are 328.4 Mpa and 22.24 GPa at fibre volume fraction of 55.62%. The flexure strength of circular ring is 80.56% of the tensile strength of plain laminate. Generally flexure strength is on lower side as compared to tensile strength. Also, the average fibre volume fraction is 71.64% of that of plain laminate, which explains for the discrepancy in the results. The average modulus of elasticity of circular ring is 93% of that of plain laminate, which is satisfactory.

More or less all the problems point towards the balloon. So balloon development must be thought of. A thin walled (~0.3mm), one piece balloon of rubber or latex material should be developed. The balloon should have cross-section and appropriate corners corresponding to the geometry of the component being fabricated.

3.8 Closure

The dimensional accuracy achieved in case of circular ring is reasonably good. Circularity is 1.3 mm and thickness variation is 0.5 mm. Thickness variation is high, but it can be controlled, by using thin, one piece balloon. The fibre volume fraction is low, but with higher curing pressure, fibre volume fraction of 55% to 60% can be achieved. The average flexure strength (at $V_f = 39.69\%$) is 80.56% of the tensile strength of laminate (at $V_f = 55.62\%$). Modulus of elasticity is close to that of plain laminate. The microscopic analysis shows satisfactory impregnation and curing of composite, with low void contents. The convex-concave surfaced ring is dimensionally stable except at sharp curvature of corner points. The microscopic analysis is satisfactory.

The circular ring with concave cross-section, a doubly curved product was also moulded well. The fibres were oriented at $[45, -45]_s$, so as to get moulded in desired shape. This product has showed, that doubly curved products can be easily moulded using Balloon Compression Moulding.

It is felt that Balloon Compression Moulding has potential to overcome some of the shortcomings of hand lay-up and bagging methods. With the application of curing pressure, denser, voids free composites can be produced at cheaper rates, as tooling is simple. As compared with the bagging method, this method is cost-effective. It can produce components having double curvature, complex and irregular shapes with reasonable accuracy.

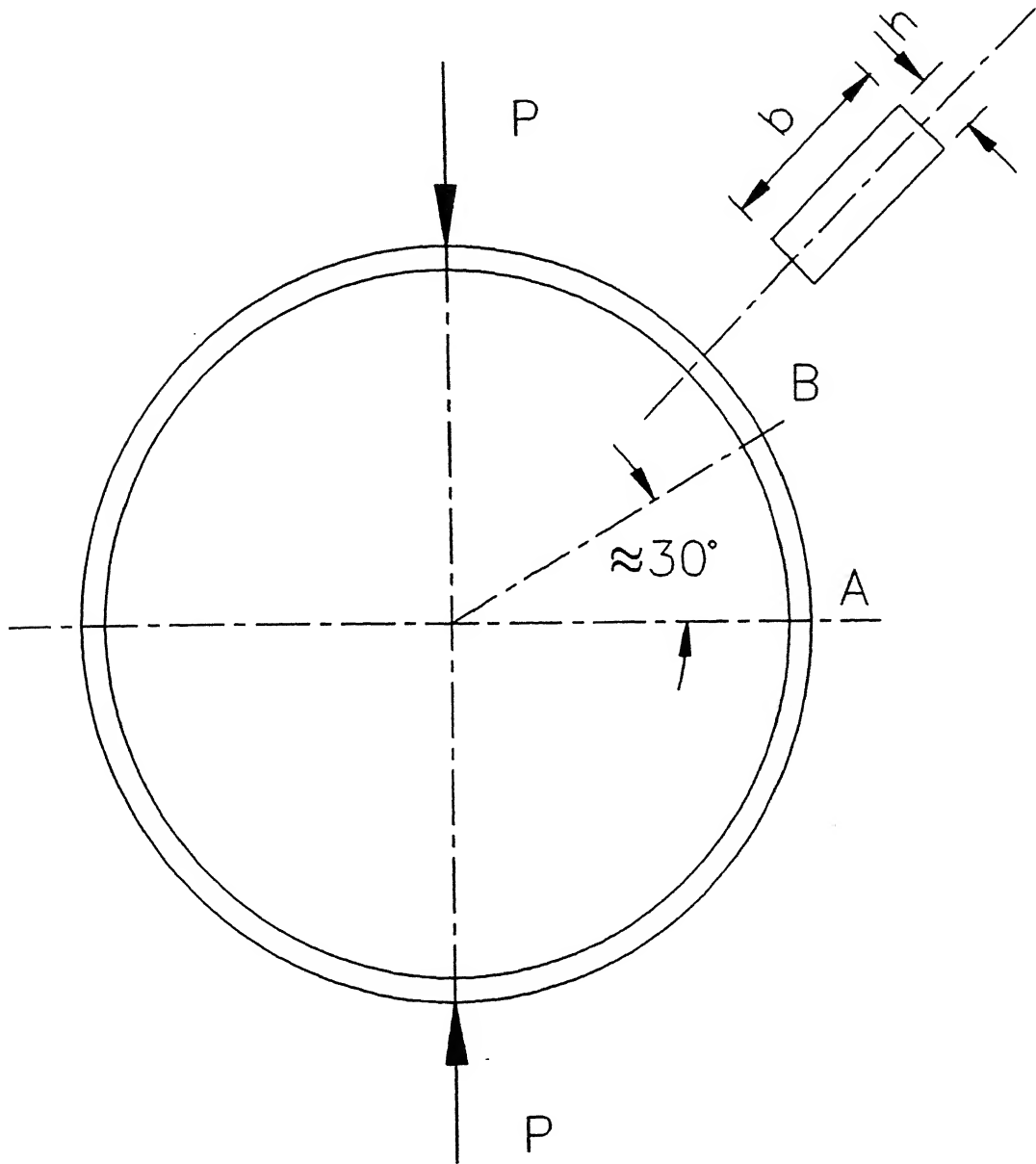


Figure 3.1 Geometry of Circular Ring Specimen

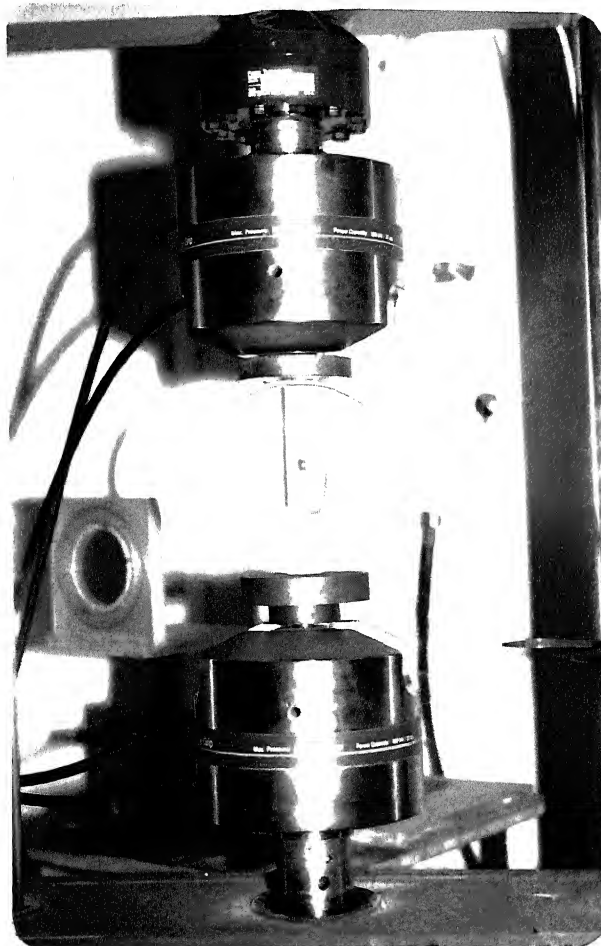


Figure 3.2 Photograph Showing Circular Ring Under Diametral Compression

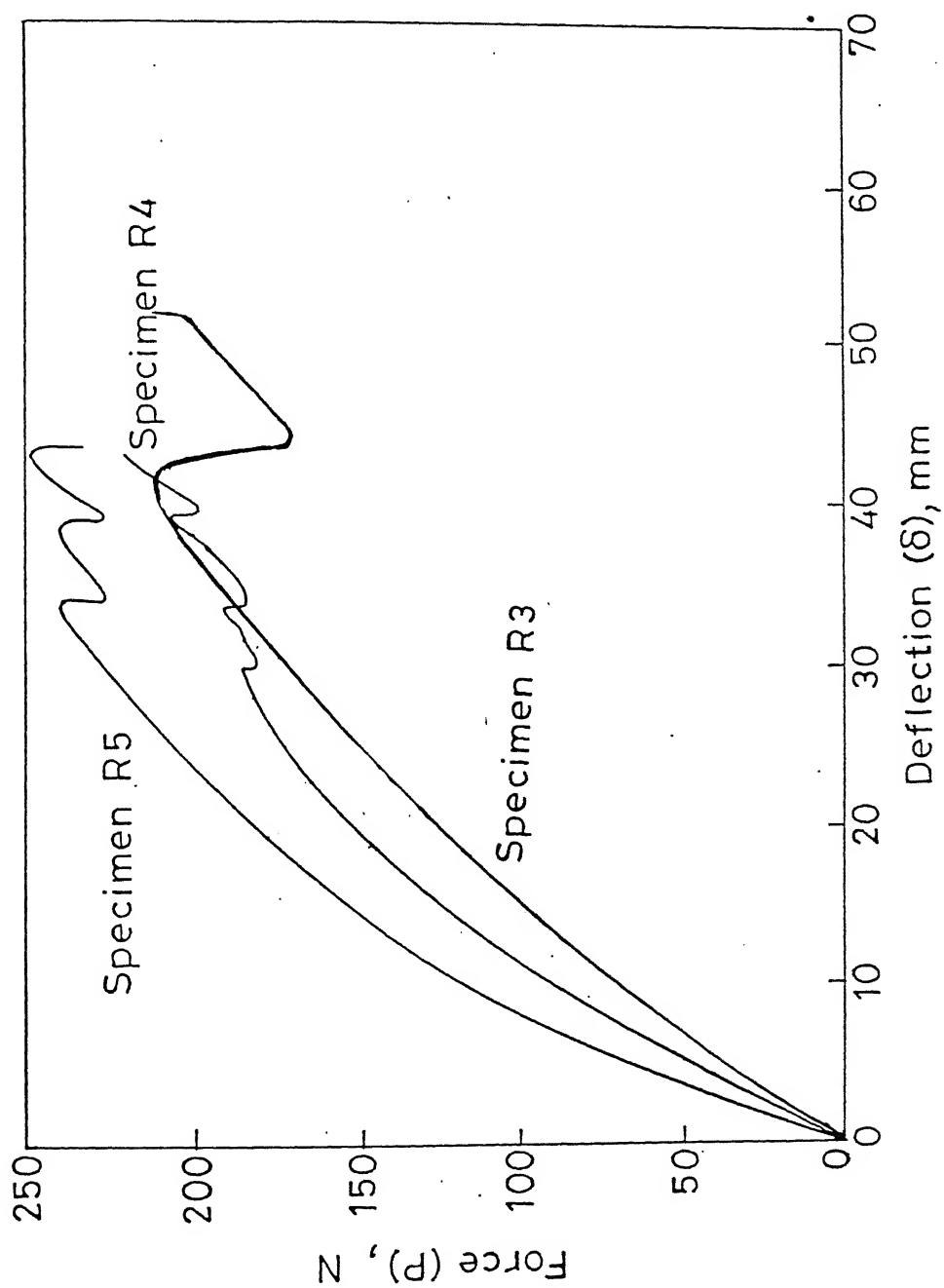
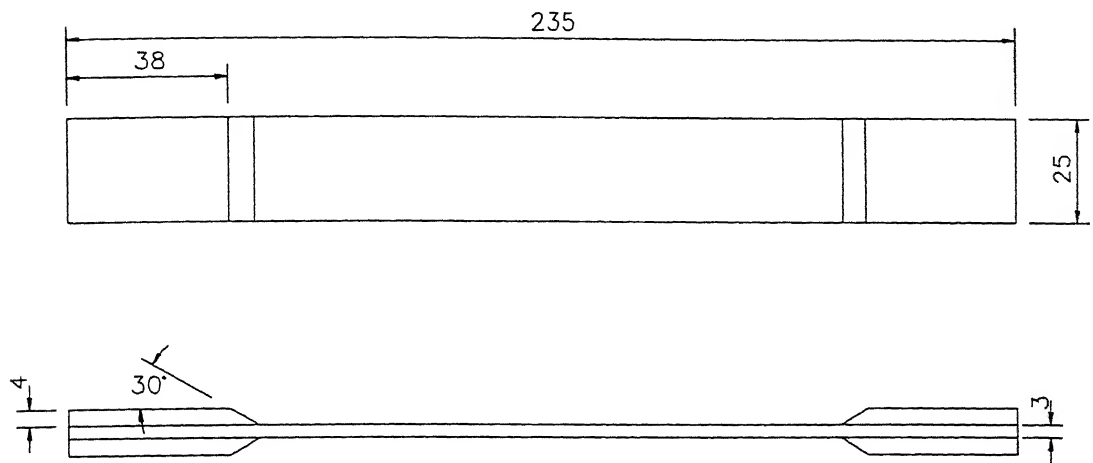


Fig. 3.3 Load deflection curve for circular ring.



All dimensions are in mm

Figure 3.4 Geometry of Tensile Specimen

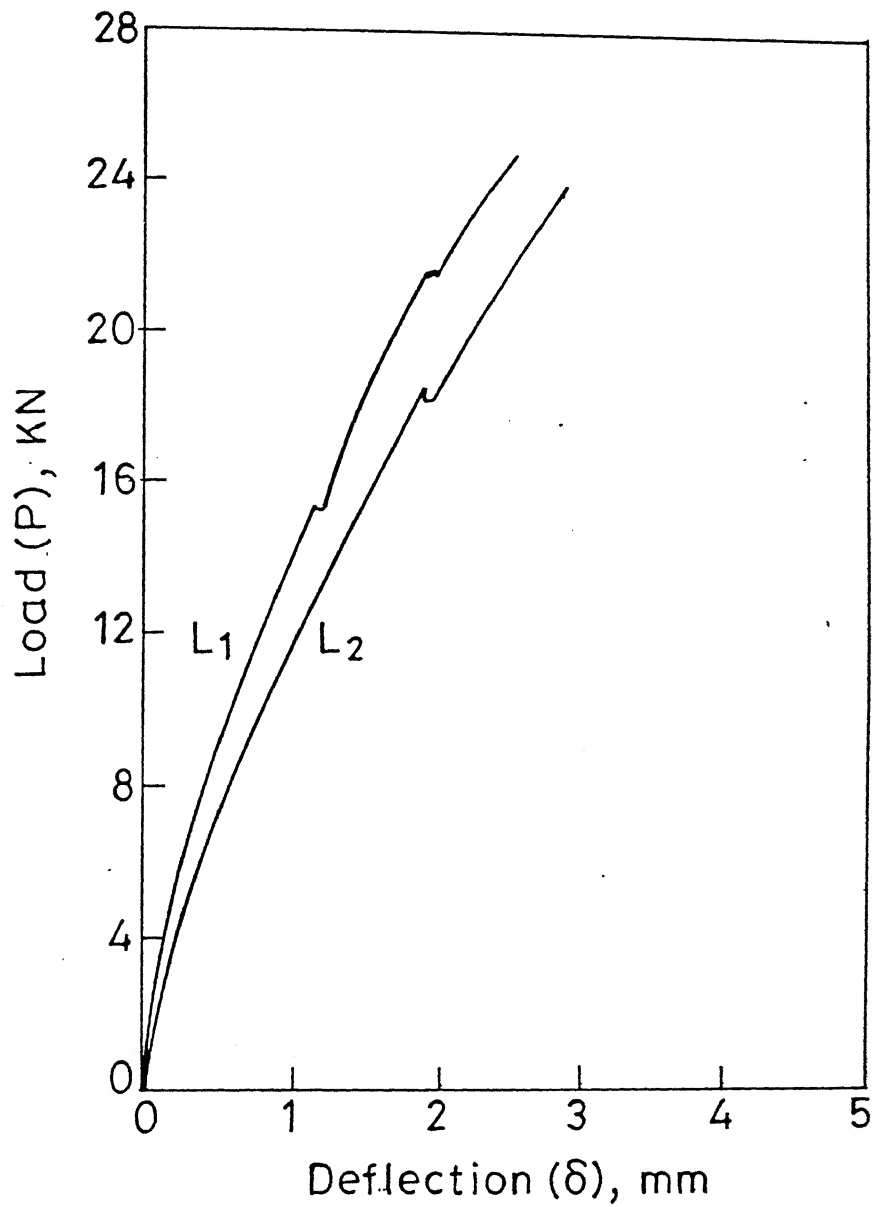
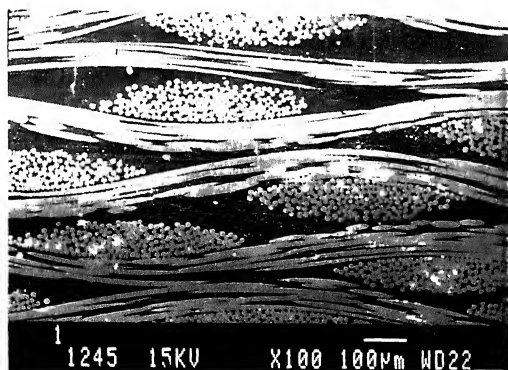
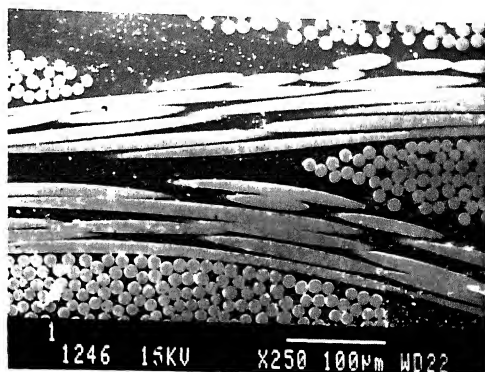


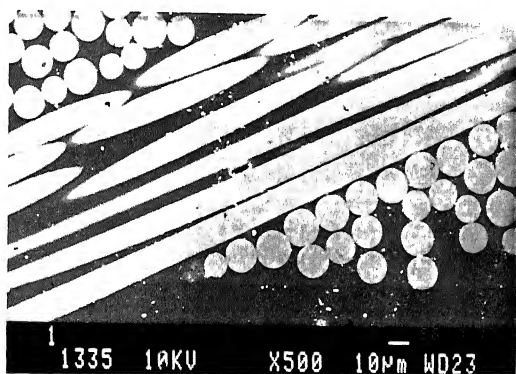
Fig.3.5 Load deflection curve for plain laminate.



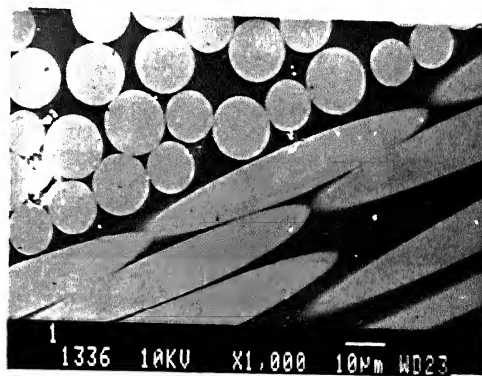
3.6(a)



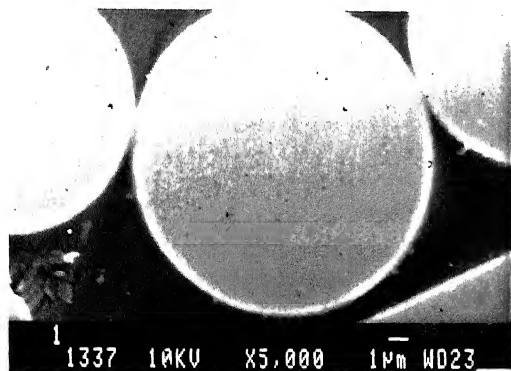
3.6(b)



3.6(c)

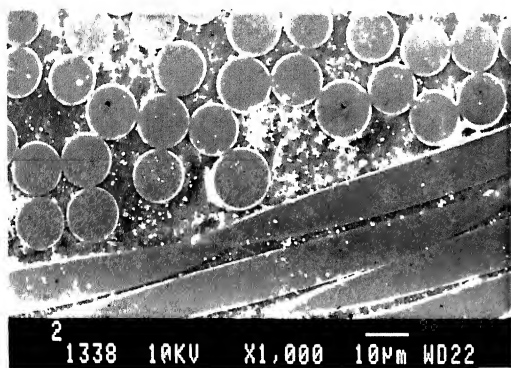


3.6(d)

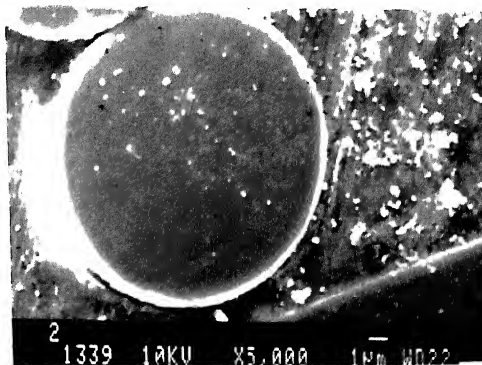


3.6(e)

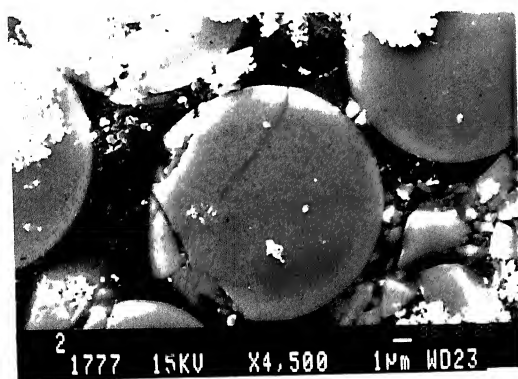
Figure 3.6 Micrographs of Virgin Specimen



3.7(a)

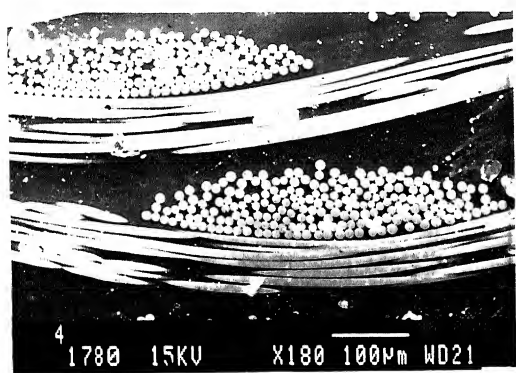


3.7(b)

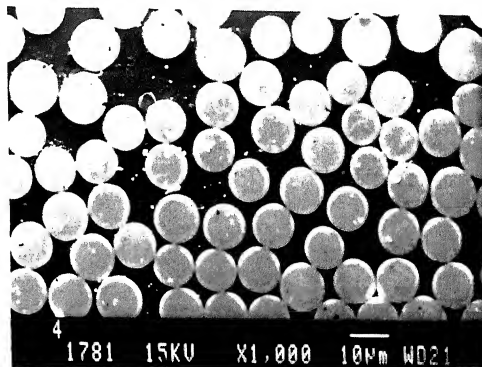


3.7(c)

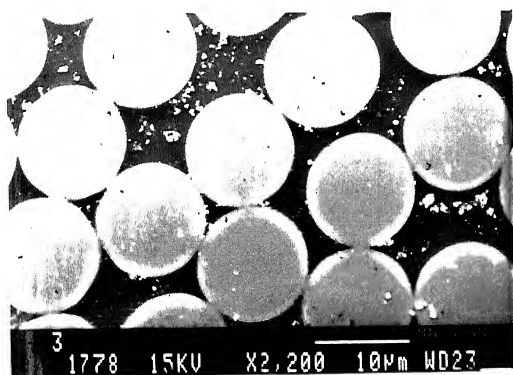
Figure 3.7 Micrographs of Fractured Specimens



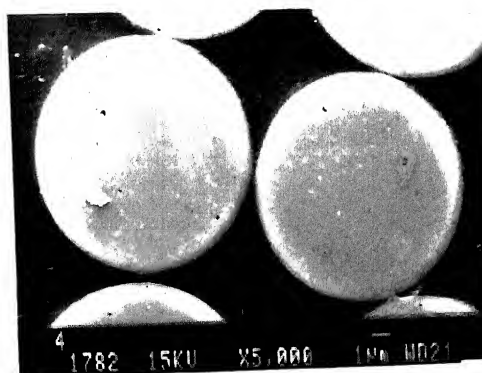
3.8(a)



3.8(b)



3.8(c)



3.8(d)

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CHAPTER 4

CONCLUSIONS AND SCOPE OF FUTURE WORK

4.1 Introduction

A new technique of fabricating polymer composite materials using Balloon Compression Moulding has been developed. The concept of moulding composite material with balloon compression was a novel one and with little bit of engineering it has worked very well. Three different products fabricated and tested, have given satisfactory results. The success of this process lies in its simple yet effective design.

Experimental results obtained are encouraging. The fibres are well impregnated. Few voids were observed, but their size is small (less than the diameter of the fibres). All dimensions were under control. The products have reasonable strength and stiffness. However fibre volume fraction was marginally lower.

The balloon proved to be a weak link in this work. Balloon made from cycle tube is not very appropriate. Balloon making technology needs to be developed in the laboratory, so as to make balloons appropriate with the geometry of the products.

4.2 Scope of Future Work

With a custom made, thin walled balloon, products having complex shapes can be fabricated. Furthermore, prepreg tapes and higher curing temperature can be used. This will add to versatility of the process. With appropriate balloon, there is unlimited variety of complex shapes, one can produce.

Some of the products, having complex geometries, which can be thought of fabricating using balloon compression moulding are gears and cycle rim.

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